



VERIFICATION

I, Dr. R. Rankers, hereby declare that all statements made in the Inspection Report F 7067/1 dated March 23, 2009, and entitled "Determination of the efficiency and resistance of ferrous sulphate as a chromate reductant" of my own knowledge are true, all statements made herein on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001, and may jeopardize the validity of the application or any patent issued thereon.

Signed: Rankers

Date: 20.10.2010

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Forschungsinstitut der Zementindustrie GmbH
Quality Assurance and Analytics

Technical Report
TB-QSA 0094/2010/F

Examination of the effectiveness of chromate reducers

Examination of the effectiveness of chromate reducers

Client:	Ferro Duo GmbH
Date of order:	February 12, 2010
Client's order no.:	-
Our order no.:	2010/0137
Project manager:	Dr. Baetzner
Person in charge:	Ms. Engel
Department:	Quality Assurance and Analytics
Issued on:	July 28, 2010
Scope of report:	4 pages

Scope of examination

The client supplied two materials, one sample designated “copperas” and one sample designated “filter salt”. The samples were examined both in the form as supplied and together in different mixtures.

In addition, different mixtures with reference cement were prepared, on the one hand by mixing and on the other by co-grinding. The reference cement was also examined in order to determine the starting chromate content.

1.1 Mixtures

Sample A: copperas
Sample B: filter salt
Sample C: copperas and filter salt in a 1:1 ratio
Sample D: copperas and filter salt in a 3:1 ratio
Sample E: copperas and filter salt in a 5:1 ratio

1.2 Dosages

Mixtures and grindings in which 0.1 M%, 0.2 M%, 0.3 M%, and 0.4 M% chromate reducer were added to the VDZ reference cement were prepared for all of the samples.

The mixtures were prepared by co-mixing the reducers with 2.5 kg cement for 30 minutes in a tumbling mixer.

Grinding occurred by co-grinding the reducers with 2.5 kg cement in a ball mill for 60 minutes.

2. Chemical tests

The content of calcium carbonate of the starting materials was determined by measuring the content of carbon dioxide in accordance with DIN EN 196-2, and the pH of the starting materials was determined in accordance with DIN EN 12176. The calcium carbonate content of the mixtures was calculated based on the contents of the starting materials and the pH was additionally determined in accordance with DIN EN 12176.

		Copperas	Filter salt	Copperas/filter salt 1:1	Copperas/filter salt 3:1	Copperas/filter salt 5:1
		A	B	C	D	E
pH		3.0	4.5	4.5	4.3	3.8
CaCO ₃	M%	<0.01	8.6	4.3	2.2	1.4

3. Examination of effectiveness

The analyses were performed, in accordance with DIN 196-10, both with and without an oxidation step. In the following, only the higher chromate contents from both determinations are stated, as required by the standard.

3.1 Effectiveness when mixed

Amount of reducer added		Reference cement	Copperas	Filter salt	Copperas/filter salt 1:1	Copperas/filter salt 3:1	Copperas/filter salt 5:1
			A	B	C	D	E
	0.0 M%	15.1 ppm					
0.7 kg/t/ppm	0.1 M%		0.2 ppm	10.0 ppm	2.2 ppm	0.3 ppm	0.4 ppm
1.3 kg/t/ppm	0.2 M%		<0.1 ppm	5.3 ppm	0.1 ppm	0.1 ppm	0.2 ppm
2.0 kg/t/ppm	0.3 M%		<0.1 ppm	0.5 ppm	<0.1 ppm	0.2 ppm	0.1 ppm
2.7 kg/t/ppm	0.4 M%		<0.1 ppm	<0.1 ppm	<0.1 ppm	<0.1 ppm	0.1 ppm

An amount of 0.7 kg reducer added per ppm chromate content and t cement is sufficient for the copperas examined in order to achieve a reduction that is almost complete. Three times as much must be used for the filter salt. For the mixtures of both materials, an addition of 1.3 kg/t/ppm reducer results in a reduction that is almost complete.

3.2 Effectiveness when ground

Amount of reducer added		Reference cement	Copperas	Filter salt	Copperas/ filter salt 1:1	Copperas/ filter salt 3:1	Copperas/ filter salt 5:1
			A	B	C	D	E
	0.0 M%	15.1 ppm					
0.7 kg/t/ppm	0.1 M%		3.5 ppm	9.5 ppm	2.8 ppm	0.1 ppm	<0.1 ppm
1.3 kg/t/ppm	0.2 M%		1.2 ppm	2.6 ppm	<0.1 ppm	<0.1 ppm	<0.1 ppm
2.0 kg/t/ppm	0.3 M%		<0.1 ppm	<0.1 ppm	<0.1 ppm	<0.1 ppm	<0.1 ppm
2.7 kg/t/ppm	0.4 M%		0.1 ppm	<0.1 ppm	<0.1 ppm	<0.1 ppm	<0.1 ppm

In the case of co-grinding, at least 2.0 kg reducer per ppm chromate content and t cement must be added of both - the copperas and the filter salt examined - in order to achieve a complete reduction. If an amount of 1.3 kg/t/ppm copperas is added, at least a residual chromate content of smaller than 2 ppm is achieved. However, when both materials are mixed, an almost complete reduction is achieved in sample C if 1.3 kg/t/ppm reducer is added and in samples D and E if only 0.7 kg/t/ppm reducer is added.

Forschungsinstitut der Zementindustrie GmbH
Department Quality Assurance and Analytics

ppa. Dr. Silvan Baetzner

pp. Dr. Gerhard Spanka

Further information, e.g. on measurement devices, measurement methods, measurement uncertainties and other method characteristics, can be provided on request. Unless specified differently by the client, the samples will be discarded four weeks after the report has been drawn up.



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